

CLEAR EMULSIFIERS WITH MICROEMULSIFIED PERFUME OILS

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CLEAR EMULSIFIERS WITH MICROEMULSIFIED PERFUME OILS

[Klare Weichspüler mit mikroemulgierten Parfümölen]

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The following data are taken from documents submitted by the applicant.

Description

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This invention concerns clear, aqueous, low- to high-concentration, perfume-containing softeners for textile softening. In particular, the invention concerns clear aqueous softeners that are suitable for use in the rinse cycle of household washing machines and that give treated textiles excellent softness, antistatic properties, easy ironing, good rewettability and a long-lasting fragrance.

Softener compositions for rinse bath treatment are widely described in the prior art. Usually, these compositions contain a cationic quaternary ammonium compound that is dispersed in water as the active agent. In each case according to the content of active substance in the end softener composition one speaks of dilute, ready-to-use products (active substance contents under 7 wt%), or the so-called concentrates (active substance contents over 7 wt%). Because of the lower volume and simultaneously reduced packaging and transport costs, textile softener concentrates have advantages from the ecological standpoint and have increasingly

* [Numbers in the right margin indicate the pagination in the original foreign text.]

gained acceptance on the market. Because of the incorporation of cationic compounds, which only have low solubility in water, the usual softener compositions are in the form of dispersions, have a milky/cloudy appearance, and are not transparent. However, for reasons of product aesthetics, it can be desirable to make available to the consumer transparent clear softeners that visually stand apart from the known products.

The production of clear softeners and the problems connected with their production are also widely described in the prior art. For example, European Patent Application EP-A-0 404 471 (Unilever) describes isotropic liquid textile softener compositions that contain at least 20 wt% softener and at least 5 wt% of a short-chain organic acid.

Clear textile softener compositions with high contents of solvent are also known, where the softening substances used here are, for example, quaternized ester ammonium compounds ("ester quats") with unsaturated, branched or short-chain alkyl residues. Such systems have the disadvantage that agglomerates, which are absorbed onto the fibers and lead to spots and reduced softness there can form. Also, the storage stability and cold resistance of such agents is frequently unsatisfactory, so that these agents become thick or are subject to precipitation or phase separation between 18°C and 4°C.

To solve these problems, WO 97/03 169 (Procter & Gamble) proposes the use of less than 40 wt% solvents that have a ClogP value between 0.15 and 0.64. The active substances described in this document have unsaturated or relatively short (C₁₂₋₁₄) alkyl chains and are contained in the agents in amounts from 2-80 wt%, preferably from 13-75 wt%, and especially from 17-70 wt%. To obtain clear softeners that contain 2-10 wt% active substance, it is necessary to employ a particular production process that requires a premix of ester quat, solvents and perfume, since the agents otherwise remain cloudy.

New ester quats are described in DE 195 39 846 (Henkel). The cationic surfactants disclosed there are reaction products of triethanolamine with mixtures of fatty acids and dicarboxylic acids. In this document hair rinses, shampoos, shower gels and washing lotions are mentioned as areas for use of these new substances. To produce these new ester quats, trialkanolamines are reacted with a mixture of fatty acids and dicarboxylic acids, preferably in mol ratios from 1:1 to 10:1, and the resulting esters are quaternized in a substantially known way.

The use of the ester quats described in DE 195 39 846 C1 to produce water-clear softeners is described in Research Disclosure, May 1997, No. 39729 (disclosed anonymously). This research disclosure discloses usage concentrations for the ester quats between 5 and 15 wt%. Nothing is mentioned in this document regarding the addition of fragrance to the agents.

With all of the clear rinses or methods for producing them that are described in the prior art, the production of low-concentration products is more costly than the production of

concentrates. In addition, the incorporation of fragrances in some cases presents considerable problems, since many perfumes do not dissolve in the formulations at all or soon separate back out. The incorporation of emulsifiers in the clear products before the addition of the perfume does not satisfactorily this problem, since high emulsifier concentrations are necessary for an acceptable storage stability.

Perfume oil concentrates in the form of transparent aqueous microemulsions have been described in the prior German Patent Application DE 196 24 051.4 (Henkel). The microemulsions disclosed there contain 10-50 wt% perfume oil, 1-10 wt% of a co-oil component, 1-30 wt% of an emulsifier of the type of the alkyl polyglycosides as well as optionally up to 10 wt% of a nonionic or cationic coemulsifier, and they have particle sizes between 10 and 100 nm, where the weight ratio of perfume oil to co-oil component is 10:1 to 2:1.

The present invention now had the task of making available clear, aqueous, low concentration to high concentration, perfume-containing softeners for textile softening that do not have these problems. In particular, clear softeners that have excellent storage stability even without the use of high concentrations of emulsifier and that are additionally characterized by excellent fragrance properties, are to be made available.

It was now found that clear, low concentration to high concentration softeners that contain perfume can be produced with the ester quats described in DE 195 39 846 if the perfume is incorporated as a perfume oil microemulsion.

The object of the invention is a clear aqueous softener containing, in each case with respect to the total agent,

- a) 2-60 wt% of a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids, which was optionally alkoxyated and then quaternized in a substantially known way,
- b) 0.5-20 wt% of a perfume oil microemulsion with a droplet size between 10 and 100 nm, which in turn contains 10-50 wt% perfume oil, 1-10 wt% of a co-oil component, 1-30 wt% of an emulsifier of the type of the alkyl polyglycosides and optionally up to 10 wt% of a nonionic or cationic coemulsifier, where the weight ratio of perfume oil to co-oil component is 10:1 to 2:1, and
- c) water or an aqueous solution of other active agents and auxiliary substances as the remainder.

The clear aqueous softeners in accordance with the invention contain the so-called ester quats as textile softening active substance. While there are a large number of possible compounds from this class of substances, in accordance with the invention ester quats that can be produced by the reaction of trialkanolamines with a mixture of fatty acids and dicarboxylic acids,

optionally subsequent alkoxylation of the reaction product, and quaternization in a substantially known way, as described in DE 195 39 846 are used.

The ester quats produced in this way are excellently suitable for the production of clear, aqueous, low- to high-concentration softeners that together with a perfume oil microemulsion and optionally other components produce clear, storage-stable and extremely effective perfumed softeners in accordance with the invention. Since in each case according to the choice of trialkanolamine, fatty acids and dicarboxylic acids, as well as the quaternization agent, a large number of suitable products can be produced and can be used in the agents in accordance with the invention, a description of the ester quats that are to be used in accordance with the invention by means of the way of producing them is more precise than giving a general formula.

The said components, which react with each other to form the ester quats that are to be used, can be added together in varying ratios. Preferred within the scope of this invention are clear softeners in which a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids in a mol ratio of 1:10 to 10:1, preferably 1:5 to 5:1, which have been optionally alkoxyated and then quaternized in a substantially known way, is contained in amounts from 2-60, preferably 3-35, and especially 5-30 wt%. Especially preferred here is the use of triethanolamine, so that other preferred clear softeners of this invention contain a reaction product of triethanolamine with a mixture of fatty acids and dicarboxylic acids in a mol ratio of 1:10 to 10:1, preferably 1:5 to 5:1, which were optionally alkoxyated and then quaternized in a substantially known way, in amounts from 2-60, preferably 3-35, and especially 5-30 wt%.

All of the acids obtained from vegetable or animal oils and fats can be used as fatty acids in the reaction mixture for production of the ester quats. Here it is also absolutely possible to use a fatty acid that is not solid at room temperature, i.e., that is paste like liquid, as the fatty acid in the reaction mixture. The fatty acids, independent of their aggregate state, can be saturated or monounsaturated to polyunsaturated. Of course, it is possible to use not only "pure" fatty acids, but rather also the technical fatty acid mixtures obtained in the decomposition of fats and oils, where again these mixtures are clearly preferred from the economic standpoint. Thus, it is possible to use in the reaction mixtures for preparation of the ester quats for the clear aqueous softeners in accordance with the invention, for example, individual species or mixtures of the following acids: caprylic acid, pelargonic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, octadecan-12-olic acid [sic], arachic acid, behenic acid, lignoceric acid, cerotic acid, melissic acid, 10-undecenoic acid, petrocelic acid, petroselaidic acid, oleic acid, elaidic acid, ricinoleic acid, linolaidic acid, α - and β -eleostearic acid, gadoleic acid, erucic acid, brassidic acid. Of course, the fatty acids with uneven numbers of carbon atoms can also be used, for example undecanoic acid, tridecanoic acid, pentadecanoic acid, heptadecanoic acid, nonadecanoic acid, heneicosanoic acid, tricosanoic acid, pentacosic acid, heptacosanoic acid.

The use of fatty acids of formula I in the reaction mixture for the preparation of the ester quats is preferred within the scope of this invention, so that preferred clear softeners contain a reaction product of trialkanolamines with a mixture of fatty acids of formula I,



in which $\text{R}^1\text{-CO-}$ stands for an aliphatic, linear or branched acyl residue with 6-22 carbon atoms and 0 and/or 1, 2, or 3 double bonds, and dicarboxylic acids in a mol ratio of 1:10 to 10:1, preferably 1:5 to 5:1, which were optionally alkoxylated and then quaternized in a substantially known way, in amounts from 2-60, preferably 3-35, and especially 5-30 wt%.

Possibilities as dicarboxylic acids that are suitable for preparation of the ester quats to be used in the agents in accordance with the invention are above all saturated or mono- or polyunsaturated α - ω -dicarboxylic acids. Examples that may be mentioned here are the saturated species oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, undecanoic and dodecanoic acids, brassylic acid, tetra- and pentadecanoic acids, thapsic acid as well as hepta-, octa- and nonadecanoic acids eicosanoic and heneicosanoic acids as well as phellogenic acid. Preferably used in the reaction mixture here are dicarboxylic acids that have the general formula II, so that clear softeners are preferably those that contain a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids of formula II



in which X stands for an optionally hydroxy-substituted alkylene group with 1-10 carbon atoms, in a mol ratio of 1:10 to 10:1, preferably 1:5 to 5:1, which were optionally alkoxylated and then quaternized in a substantially known way, in amounts from 2-60, preferably 3-35, and especially 5-30 wt%.

Among the large number of ester quats that can be produced and used in accordance with the invention again those in which the alkanolamine is triethanolamine and the dicarboxylic acid is adipic acid are tried and true. Thus, within the scope of this invention clear softeners that contain a reaction product of triethanolamine with a mixture of fatty acids and adipic acid in a mol ratio of 1:5 to 5:1, preferably 1:3 to 3:1, which was then quaternized in a substantially known way, in amounts from 2-60, preferably 3-35, and especially 5-30 wt%, are especially preferred.

The perfume oil microemulsions that are used in agents in accordance with the invention, which enable intensive perfuming and transfer of fragrance to the treated textiles, without the

agents losing their transparency, have particle sizes between 10 and 100 nm and contain 10-50 wt% perfume oil, 1-10 wt% of a co-oil component, 1-30 wt% of an emulsifier of the type of the alkyl polyglycosides, and optionally up to 10 wt% of a nonionic or cationic coemulsifier, where the weight ratio of perfume oil to co-oil component is 10:1 to 2:1.

Individual odorant compounds, for example the synthetic products of the type of the esters, ethers, aldehydes, ketones, alcohols, and hydrocarbons, can be used as perfume oils or fragrances. However, mixtures of different odorants that together produce a pleasant odor note are preferred. Such perfume oils are also natural odorant mixtures such as are obtainable from plant sources, for example pine, citrus, jasmine, patchouli, rose, or ylang-ylang oil. Likewise suitable are muscatel, sage oil, chamomile oil, clove oil, melissa oil, mint oil, cinnamon leaf oil, linden flower oil, juniper berry oil, vetiver oil, olibanum oil, galbanum oil, and labdanum oil, as well as orange petal oil, neroliol, orange peel oil, and sandalwood oil.

In principle, all of the natural and synthetic oil components that are suitable for cosmetics are suitable as co-oil component, for example those of the type of the paraffin oils, the vegetable oils (triglyceride oils), liquid waxes, jojoba oil, the synthetic fatty acid and fatty alcohol esters, the dicarboxylic acid esters, the esters of diols and polyols, the linear and branched fatty alcohols, and the dialkyl ethers.

Dialkyl ethers with a total of 12-24 carbon atoms are preferably used as co-oil component in the perfume oil microemulsions in an amount of at least 0.5 wt%, so that a preferred clear softener contains a perfume oil microemulsion in which a dialkyl ether with a total of 12-24 C atoms is contained as co-oil component in an amount of at least 0.5 wt%, in amounts from 0.5-20, preferably 1-10, and especially 1.5-7.5 wt%. Also, mixtures of dialkyl ethers with primary alcohols can be used as co-oil component in the perfume oil microemulsions. Preferred within the scope of this invention are clear softeners that contain a perfume oil microemulsion in which a mixture of a dialkyl ether with a total of 12-24 C atoms and a monohydric primary alcohol with 12-36 C atoms is contained as the co-oil component in amounts from 0.5-20, preferably 1-10, and especially 1.5-7.5 wt%.

Although the preparation of the perfume oil microemulsions when the dialkyl ether is used as co-oil component can also be managed without other co-emulsifiers, it can be useful to add a co-emulsifier. Lipophilic, nonionic emulsifiers or cationic emulsifiers are suitable as co-emulsifiers. For example, fatty acid polyol partial esters of fatty acids with 10-18 C atoms and a polyol with 2-6 C atoms and 2-6 hydroxyl groups can be used as lipophilic nonionic coemulsifiers. Examples of such coemulsifiers are, for example, sorbitan monooleate, glycerol monooleate, methyl glucoside monomyristate, propylene glycol monopalmitate, and the addition products of 1-4 mol ethylene oxide to such polyol partial esters.

Preferably alkyl oligoglycosides are used as nonionic emulsifiers for preparation of the microemulsions. Alkyl oligoglycosides, their preparation, and their use as surface-active agents are known, for example, from DE 19 43 689 A1 or DE 38 27 543 A1. With regard to the glycoside residue both monoglycosides in which a sugar residue is glycosidically bonded to the fatty alcohol, and oligomer glycosides with an average degree of oligomerization of up to about 2 are suitable. Suitable alkyl oligoglycosides are ones of the formula $RO-(G)_x$, in which RO means the aliphatic linear residue of a primary fatty alcohol with 8-22, preferably 10-16 C atoms, and $(G)_x$ is an oligoglycoside residue with an average degree of oligomerization x from 1-2. The commercial alkyl oligoglycosides contain the glucoside residue as glycoside residue. The weight ratio of perfume oil (A) to alkyl glycoside (C) is preferably in the range from 0.5:1 up to 2:1.

The alkyl polyglycoside is used as emulsifier, preferably in amounts from 1-30 wt%. Preferred perfume oil microemulsions in this case have a weight ratio of perfume oil to alkyl polyglycoside that lies in the range from 0.5:1 to 2:1. A clear softener that is preferred within the scope of this invention contains a perfume oil microemulsion in which the weight ratio of perfume oil to alkyl polyglycoside lies in the range from 0.5:1 up to 2:1, in amounts from 0.5-20, preferably 1-10, and especially 1.5-7.5 wt%.

Suitable cationic coemulsifiers are, for example, quaternary ammonium surfactants, for example cetyltrimethylammonium chloride, benzalkonium chloride, distearyldimethylammonium chloride, and especially readily biodegradable "ester quats." Besides the said quaternary ammonium salts, however, it is also possible to use other known cationic surfactants and emulsifiers as coemulsifiers in the scope of this invention.

In addition to components (a) and (b), which by themselves already yield a complete textile softener, the agents in accordance with the invention can contain other ingredients, which further improve the technical application and/or aesthetic properties of the textile softener. Within the scope of this invention preferred agents contain, in addition to components (a) and (b), one or more substances from the group consisting of electrolytes, nonaqueous solvents, pH adjusting agents, fragrances, perfume carriers, fluorescence agents, dyes, hydrotopes, foam inhibitors, silicone oils, antiredeposition agents, thickeners, enzymes, optical brighteners, graying inhibitors, antishrink agents, antcrease agents, dye transfer inhibitors, antimicrobial agents, germicides, fungicides, antioxidants, corrosion inhibitors, antistatic agents, ironing aids, waterproofing and impregnation agents, swelling and nonslip agents, as well as UV absorbers.

A large number of quite varied salts can be used as electrolytes from the group of the inorganic salts. Preferred cations are the alkali and alkaline earth metals, while preferred anions are the halides and sulfates. From the technical manufacturer's standpoint the use of NaCl or $MgCl_2$ is preferred in the agents in accordance with the invention.

Nonaqueous solvents that can be used in the agents in accordance with the invention

derive, for example, from the group of mono- or polyhydric alcohols, alkanolamines or glycol ethers, provided they are miscible with water in the given concentration range. Preferably, the solvents are chosen from ethanol, n-propanol, or isopropanol, butanols, glycol, propanediol or butanediol, glycerol, diglycol, propyl or butyl diglycol, hexylene glycol, ethylene glycol methyl ether, ethylene glycol ethyl ether, ethylene glycol propyl ether, ethylene glycol mono-n-butyl ether, diethylene glycol methyl ether, diethylene glycol ethyl ether, propylene glycol methyl, ethyl or propyl ethers, dipropylene glycol methyl or ethyl ethers, methoxy, ethoxy or butoxy triglycol, 1-butoxyethoxy-2-propanol, 3-methyl-3-methoxybutanol, propylene glycol t-butyl ether and mixtures of these solvents.

In order to bring the pH value of the agents in accordance with the invention into the desired range it may be necessary to add pH adjusting agents. Here all of the known acids or alkalis can be used, provided their use is not forbidden for technical application or ecological reasons or for reasons of consumer protection. Usually the amount of these pH adjusting agents does not exceed 1 wt% of the total formulation.

In order to improve the aesthetic impression of the agents in accordance with the invention they can be colored with suitable dyes. Preferred dyes, the choice of which does not offer any difficulty to the specialist, have a high storage stability and insensitivity to the other ingredients of the agents and to light and also do not have pronounced substantivity to textile fibers, in order not to discolor them.

Possibilities as foam inhibitors that can be used in the agents in accordance with the invention are, for example, soaps, paraffins or silicone oils, which can optionally be applied to carrier materials. Suitable antiredeposition agents, which were also called soil repellents, are, for example, nonionic cellulose ethers like methylcellulose and methylhydroxypropylcellulose with a fraction of methoxy groups from 15-30 wt% and hydroxypropyl groups from 1-15 wt%, in each case with respect to the nonionic cellulose ether, and the polymers of phthalic acid and/or terephthalic acid that are known from the prior art, or their derivatives, especially polymers of ethylene terephthalates and and/or polyethylene glycol terephthalates, or anionically and/or nonionically modified derivatives of these compounds. Of these, the sulfonated derivatives of the phthalic acid and terephthalic acid polymers are especially preferred.

Thickeners can be added to the agents in accordance with the invention to increase the viscosity or to improve the thixotropic properties of gels. Thickeners are frequently also called swelling agents and mostly are organic high-molecular substances that can absorb liquids (mostly water) swelling and in the end converting to viscous true or colloidal solutions in doing so. Examples of such agents are polyacrylic acid or acrylic acid copolymers, such as are sold, for example, by the Goodrich company under the trade name Carbopol®, starch or cationically modified starch.

Possibilities as enzymes are in particular ones from the classes of the hydrolases like the proteases, esterases, lipases or lipolytically enzymes, amylases, cellulases or other glycosyl hydrolases, and mixtures of the said enzymes. All of these hydrolases contribute to the removal of spots such as protein-, oil- or starch-containing spots and graying in the wash. Cellulases and other glycosyl hydrolases moreover can contribute to the removal of pilling and microfibrils in order to maintain color and to increase the softness of the textile. Also, oxidoreductases can be used for bleaching or to inhibit dye transfer. Especially well suited are enzymic active agents obtained from bacterial strains or fungi like *Bacillus subtilis*, *Bacillus licheniformis*, *Streptomyces griseus*, and *Humicola insolens*. Proteases of subtilisin type and especially proteases obtained from *Bacillus lentus* are preferably used. Enzyme mixtures, for example of protease and amylase or of protease and lipase or lipolytically acting enzymes, or protease and cellulase or cellulase and lipase or lipolytically acting enzymes, or protease, amylase and lipase or lipolytically acting enzymes, or protease, lipase or lipolytically acting enzymes and cellulase, but especially protease and/or lipase-containing mixtures or mixtures with lipolytically acting enzymes are of particular interest. Examples of such lipolytically acting enzymes are the well known cutinases. Also, peroxidases or oxidases have proved to be suitable in some cases. Among the suitable amylases are in particular α -amylases, isoamylases, pullulanases, and β -glucosidases, which are also called cellobiases, or mixtures of these substances. Since different cellulase types differ in their CMCase and avicelase activities, the desired activities can be established through the controlled mixing of the cellulases. The enzymes can be adsorbed on carriers or embedded in coating substances in order to protect them against premature decomposition. The amount of enzymes, enzyme mixtures, or enzyme granulates can be, for example, about 0.1-5 wt%, preferably 0.12 to about 2 wt%.

Optical brighteners (the so-called "white toners") can be added to the agents in accordance with the invention in order to remedy graying and yellowing of treated textiles. These substances are absorbed onto the fibers and bring about a brightening and simulated bleaching activity, by converting invisible ultraviolet radiation to visible longer wavelength light, where the ultraviolet light absorbed from sunlight is radiated as pale bluish fluorescence and with the yellowish tone of the grayed or yellowed laundry produces pure white. Suitable compounds derive, for example, from the substance classes of the 4,4'-diamino-2,2'-stilbenedisulfonic acids (flavonic acids), 4,4'-distyrylbiphenyls, methylumbelliferone, coumarins, dihydroquinolinones, 1,3-diarylpyrazolines, naphthalic imides, benzoxazole, benzisoxazole and benzimidazole systems, and the pyrene derivatives that are substituted by heterocycles. The optical brighteners are usually used in amounts between 0.1 and 0.3 wt% with respect to the end agent.

Graying inhibitors [antiredeposition agents] have the job of keeping soil separated from the fibers suspended in the wash water and thus preventing the redeposition of the soil. Suitable for this use are water-soluble colloids, mostly of organic nature, for example the water-soluble salts of polymeric carboxylic acids, size, gelatins, salts of ether sulfonic acids, starch or cellulose, or salts of acid sulfuric acid esters of cellulose or starch. Also, water-soluble acid group-containing polyamides are suitable for this purpose. In addition, soluble starch preparations and other starch products than those mentioned above can also be used, for example degraded starch, aldehyde starches, and so forth. Polyvinylpyrrolidone can also be used. However, cellulose ethers like carboxymethylcellulose (Na salts), methylcellulose, hydroxyalkylcellulose and mixed ethers like methylhydroxyethylcellulose, methylhydroxypropylcellulose, methylcarboxymethylcellulose and their mixtures in amounts from 0.1 to 5 wt%, with respect to the agents, are preferably used. /6

Since textile articles, especially ones of rayon, synthetic wool, cotton and their blends, can tend to wrinkle, since the individual fibers are sensitive to bending, folding, pressing and crushing across the direction of the fiber, the agents in accordance with the invention can contain synthetic antcrease agents. Among these are, for example, synthetic products based on fatty acids, fatty acid esters, fatty acid amides, fatty acid alkylol esters, fatty acid alkylol amides, or fatty alcohols, which are mostly reacted with ethylene oxide, or products based on lecithin or modified phosphoric acid esters.

To control microorganisms the agents in accordance with the invention can contain antimicrobial agents. Here in each according to antimicrobial spectrum and mechanism of action, one distinguishes between bacteriostatics and bactericides, fungistatics and fungicides, etc. Important substances from these groups are, for example, benzalkonium chlorides, alkyl aryl sulfonates, halophenols, and phenol mercury acetate, where in the case of agents in accordance with the invention these compounds can also be omitted entirely.

In order to prevent undesired changes in the agents and/or the treated textiles due to the effects of oxygen or other oxidative processes, the agents can contain antioxidants. Among this class of compounds are, for example, substituted phenols, hydroquinones, pyrocatechols and aromatic amines, as well as organic sulfides, polysulfides, dithiocarbamates, phosphites, and phosphonates.

Improved wearing comfort can result from the additional use of antistatic agents, which are additionally added to the agents in accordance with the invention. Antistatics increase surface conductivity and thereby enable improved drainage of charges that have formed. External antistatics as a rule are substances with at least one hydrophilic molecular ligand and they produce a more or less hygroscopic film on the surface. These mostly surface-active antistatics can be divided into nitrogen-containing antistatics (amines, amides, quaternary ammonium

compounds), phosphorus-containing antistatics (phosphoric acid esters), and sulfur-containing antistatics (alkyl sulfonates, alkyl sulfates). External antistatics are described, for example, in Patent Applications FR 1,156,513, GB 873 214, and GB 839 407. The lauryl (or stearyl) dimethylbenzylammonium chlorides disclosed here are suitable as antistatics for textiles or as additives to washing agents, where a softening effect is additionally achieved.

To improve the water absorption capacity and rewettability of the treated textiles and to improve ironing of the treated textiles silicone derivatives, for example, can be used in the agents in accordance with the invention. These compounds additionally improve the rinse-out behavior of the agents in accordance with the invention due to their foam-inhibiting properties. Preferred silicone derivatives are, for example, polydialkyl- or alkylaryl siloxanes, in which the alkyl groups have one to five C atoms and are partially or entirely fluorinated. Preferred silicones are polydimethylsiloxanes, which can optionally be derivatized and then are amino functional or quaternized, or have Si-OH, Si-H and/or Si-Cl bonds. The viscosities of the preferred silicones at 25°C lies in the range between 100 and 100,000 centistokes, and the silicones can be used in amounts between 0.2 and 5 wt% with respect to the total agent.

Finally, the agents in accordance with the invention can also contain UV absorbers, which are absorbed onto the treated textiles and improve the resistance of the fibers to light. Compounds that have these desired properties are, for example, the compounds and derivatives of benzophenone with substituents in position 2 and/or position 4 that act through radiationless deactivation. Also suitable are substituted benzotriazoles, acrylates with phenyl substituents in position 3 (cinnamic acid derivatives), optionally with cyano groups in position 2, salicylates, organic Ni complexes, as well as natural substances like umbelliferone and the endogenous urocanic acid.

The preparation of the clear softeners in accordance with the invention takes place in a substantially known way by simply mixing the individual components, where the perfume oil microemulsion is prepared separately. In each case after other optional ingredients have been added in the form of aqueous solutions or as solids that have to be dissolved, it can be advantageous to add the perfume oil microemulsion as the last item and then to mix it in. The effect of high shear forces or the use of high energy mixers as are needed to produce stable traditional dispersions, is not necessary.

Examples

A perfume oil microemulsion of the following composition was prepared by intensive mixing of the components indicated in Table 1:

Table 1. Perfume oil microemulsion (wt%)

Plantacare® 220	4.0
Plantacare® 1200	16.0
Dehyquart AU 46	2.0
Cetiol OE	7.5
Perfume	2.0.0
Water	Remainder

Plantacare® 220: C₈-C₁₀ alkyl oligo(1,5)glucoside, 63% in water, commercial product of Henkel, Düsseldorf

Plantacare® 1200: C₁₂-C₁₆ alkyl oligo(1,4)glucoside, 50% in water, commercial product of Henkel, Düsseldorf

Dehyquart® AU46: Dipalmitolexyloxyethylhydroxyethylmethylammonium ethoxy sulfate, 90% in isopropanol, commercial product of Henkel, Düsseldorf

Cetiol® OE: di-n-octyl ether, commercial product of Henkel, Düsseldorf.

An adipic acid ester quat produced by the teaching of DE 195 39 846 (Example 1), which was used in low concentration and high concentration softeners, was used to produce clear softeners. The softeners E1 and E2 in accordance with the invention and the Comparison Examples V1 to V4 were in each case perfumed with the same amount of perfume, where in accordance with the invention the perfume oil microemulsion was used, while in the comparison examples the perfume was incorporated directly. The preparation of products E1 and V1 took place by mixing the adipic acid ester quat into 50°C water, stirring it for 15 min at 50°C, cooling it to 25°C and then adding the perfume oil microemulsion or perfume. The preparation of products E2 and V2 took place by mixing the adipic acid ester quat in 60°C water, adding the emulsifier and solvents, stirring for 15 min at 60°C, cooling to 25°C and then adding the perfume oil microemulsion or perfume. To confirm that the components additionally present in the perfume oil microemulsion do not have an effect on their own, but rather are effective only as microemulsion with the perfume, an emulsifier mixture analogous to the emulsifier mixture used in the perfume oil microemulsion was prepared for Comparison Examples 3 and 4 and mixed into the batch before the addition of the perfume oil. The composition of the emulsifier mixture is given in Table 2, while Table 3 shows the composition of the agents and data on stability and appearance.

Table 2. Composition of the emulsifier mixture (wt%)

Plantacare 220	13,6
Plantacare 1200	54,2
Dehyquart AU 46	6,8
Cetiol OE	25,4

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Table 3. Composition of softeners (wt%)

	E 1	V 1	V 3	E 2	V 2	V 4
Adipinsäure-Esterquat*	6,0	6,0	6,0	35,0	35,0	35,0
Parfüm	-	0,3	0,3	-	1,5	1,5
Parfümöl-Mikroemulsion	1,5	-	-	7,5	-	-
Arylpon F	-	-	-	12,0	12,0	12,0
2-Methyl-2,4-pentandiol	-	-	-	10,0	10,0	10,0
Emulgatormischung**	-	-	0,44	-	-	2,2
Wasser	Rest	Rest	Rest	Rest	Rest	Rest
Aussehen (nach Herstellung)	③ Klare Lösung	2 Phasen ④	2 Phasen ④	③ Klare Lösung	trüb ⑤	trüb ⑤
Aussehen (7 Tage Lagerung)	③ Klare Lösung	2 Phasen ④	2 Phasen ④	③ Klare Lösung	2 Phasen ④	trüb ⑤

⑥ : 80%-ig in Ethanol

⑦ Arylpon F : C₁₂₋₁₄-Fettsäurealkohol mit 2,5 Mol EO

- Key: 1 Adipic acid ester quat*
 Perfume
 Perfume oil microemulsion
 Arylpon F
 2-Methyl-2,4-pentenediol
 Emulsifier mixture**
 Water
 Appearance (after preparation)
 Appearance (7 days storage)
- 2 Remainder
 3 Clear solution
 4 2 phases

- 5 Cloudy
- 6 80% in ethanol
- 7 Arylpon F: C₁₂₋₁₄ fatty alcohol with 2.5 mol EO

Claims

1. A clear aqueous softener containing, in each case with respect to the total agent,
 - a) 2-60 wt% of a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids, which was optionally alkoxylated and then quaternized in a substantially known way,
 - b) 0.5-20 wt% of a perfume oil microemulsion with a droplet size between 10 and 100 nm, which in turn contains 10-50 wt% perfume oil, 1-10 wt% of a co-oil component, 1-30 wt% of an emulsifier of the type of the alkyl polyglycosides and optionally up to 10 wt% of a nonionic or cationic coemulsifier, where the weight ratio of perfume oil to co-oil component is 10:1 to 2:1, and
 - c) water or an aqueous solution of other active agents and auxiliary substances as the remainder.
2. A clear softener as in Claim 1, which is characterized by the fact that it contains a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids in a mol ratio of 1:10 to 10:1, preferably 1:5 to 5:1, which was optionally alkoxylated and then quaternized in a substantially known way, in amounts from 2-60, preferably 3-35, and especially 5-30 wt%.
3. A clear softener as in one of Claim 1 or 2, which is characterized by the fact that it contains a reaction product of triethanolamine.
4. A clear softener as in one of Claims 1-3, which is characterized by the fact that it contains a reaction product of trialkanolamines with a mixture of fatty acids of formula I,



in which R¹-CO- stands for an aliphatic, linear, or branched acyl residue with 6-22 carbon atoms and 0 and/or 1, 2, or 3 double bonds.

5. A clear softener as in one of Claims 1-4, which is characterized by the fact that it contains a reaction product of trialkanolamines with a mixture of fatty acids and dicarboxylic acids of formula II,



in which X stands for an optionally hydroxy-substituted alkylene group with 1-10 carbon atoms.

6. A clear softener as in one of Claims 1-5, which is characterized by the fact that it contains a reaction product of triethanolamine with a mixture of fatty acids and adipic acid in a mol ratio of , in a mol ratio of 1:5 to 5:1, preferably 1:3 to 3:1, which was then quaternized in a substantially known way.

7. A clear rinse agent as in one of Claims 1-6, which is characterized by the fact that it contains a perfume oil microemulsion in which a dialkyl ether with a total of 12-24 C atoms is contained as co-oil component in an amount of at least 0.5 wt%, in amounts from 0.5-20, preferably 1-10, and especially 1.5-7.5 wt%. /9

8. A clear softener as in one of Claims 1-7, which is characterized by the fact that it contains a perfume oil microemulsion in which a mixture of a dialkyl ether with a total of 12-24 C atoms and a monohydric primary alcohol with 12-36 C atoms is contained as co-oil component, in amounts from 0.5-20, preferably 1-10, and especially 1.5-7.5 wt%.

9. A clear softener as in one of Claims 1-8, which is characterized by the fact that it contains a perfume oil microemulsion in which the weight ratio of perfume oil to alkyl polyglycoside is in the range from 0.5:1 to 2:1.

10. A clear softener as in one of Claims 1-9, which is characterized by the fact that in addition to components (a) and (b) it contains one or more substances from the group consisting of electrolytes, nonaqueous solvents, pH adjusting agents, fragrances, perfume carriers, fluorescence agents, dyes, hydrotopes, foam inhibitors, silicone oils, antiredeposition agents, thickeners, enzymes, optical brighteners, graying inhibitors, antcrease agents, dye transfer inhibitors, antimicrobial agents, germicides, fungicides, antioxidants, corrosion inhibitors, antistatics, ironing aids, waterproofing and impregnation agents, swelling agents and nonslip agents, and UV absorbers.